DEVICE AND METHOD FOR TREATING PLASTIC MATERIAL

The invention relates to a device according to the
precharacterising portion of claim 1 as well as to a
method with the characteristics of the
precharacterising portion of claim 8.

Such a method has for example been disclosed in EP-A-0
712 703. When such a method is implemented in practical
applications, the finished product has some significant
deficiencies:

- the product is hydrolysed, i.e. partly decomposed
  and thus its quality is insufficient;
- the finished product contains a relatively high
  dust fraction which not only means loss of
  material but also increased cost per unit of
  weight of the end product; and
- the degree of crystallisation of the finished
  product is quite variable and the amorphous
  fraction is relatively high.

It is thus the object of the invention to create a
product, e.g. bottle granulate made of PET or polyester
material for tyre cord etc., of improved quality with a
low dust fraction and improved crystallinity. According
to the invention this is achieved by the characterising
portions of claim 1 or of claim 8.

The invention is based on the recognition that up to
now, the production parameters have been selected in a
somewhat carefree manner, with a bias to quick
production. It has been found for example that slower

heating during crystallisation with lower temperatures
allows more gentle and economical production where in
some cases even air can be used as a treatment medium
instead of the normally used nitrogen.

It has also been found that the relatively high gas
temperatures exceeding 195 °C hitherto applied not only
lead to product decomposition and thus to a drop in
quality, but also cause excessive heat loss in the
apparatus hitherto used. It is for example known from
US-A-5,119,570 to carry out precrystallisation and
crystallisation in separate apparatus. However,
separate apparatus also means a relatively high
surface-to-volume ratio which promotes heat loss. While
in the above-mentioned EP-A, precrystallisation and
crystallisation are carried out in a mutual device,
this does not result in optimisation either.

From these findings, the characterising portion of
claim 1 was developed which results in a compact and
economical design which at the same time results in
minimal heat loss. The small heat losses also result in
the conditions of crystallisation being better able to
be kept under control, so that from this point of view
too, there is no compulsion to apply high treatment
temperatures. There are above all advantages in that
the gas throughput for the compartments from a single
gas source can be smaller than was the case so far and
in that the overall height of the device itself can be
kept lower, thus resulting in savings of space and
cost. While a crystalliser with a rotation-symmetrical
housing is known from CH-A-665 473, it is not designed
or suitable for carrying out precrystallisation and
crystallisation, i.e. it requires an additional device
for a separate crystallisation step.

In the above mentioned EP-A the path of the plastic
material flowing through the device is such that a high
throughput of air is required so as to generate an
aggregate fluidisation, and that the material is thrown
above a free space situated above a partition wall,
into the next compartment. By contrast, the invention
preferably provides for the path to meander by
arranging free space or free spaces and discharge
aperture(s) at various levels in longitudinal section
through the treatment space. While the known throwing-
over results in significant differences in dwell times
of the individual material components, with the measure
according to the invention there is better control over
the dwell time. It has been shown that in this way an
excellent crystallisation degree with negligible
amorphous fraction can be achieved. By contrast, in the
case of a meandering path, the quantity of air flowing
through can be reduced. In addition, in the case of
free spaces or apertures located at the bottom, there
is a certain division of the treated material according
to its density even in a fluidised bed, with material
of increased relative density being more likely to be
in the bottom region. While the differences in density
between amorphous and crystallised material in the
fluidised bed are not very large, the fact that the
denser material near the bottom is already crystallised
out to a larger degree than the material fluidising
further up, does have some significance. Consequently,
from the lower free spaces, predominantly the material
which has undergone certain crystallisation progress,
is conveyed to the next compartment (or to the
discharge aperture).

According to the above-mentioned findings, the method
according to the invention starts off at relatively low
temperatures which treat the material gently. While at
first this requires somewhat more time, it does however

bring about the preconditions for material of improved
quality. The time lost in comparison to that of the
state of the art can be saved by the subsequent shorter
treatment time during preheating or precondensation.
Preferably a fluidised bed design is used, so that
there is no need for any agitator devices within the
treatment space (compare US-A-4,064,112 and 4,161,578),
for it has been shown that said agitator devices cause
significant losses due to strong dust formation. It
must be pointed out that separation of condensation
into precondensation and postcondensation with
different treatment conditions has already been
disclosed in US-A-3,756,990 and that it is also the
preferred way of implementing the method according to
the present invention. This is also reflected in the
final temperature of the material of at least 185 °C,
preferably at least 200 °C, in particular approx. 220
°C.

In the aforesaid, among other things, the unevenness of
the quality of the finished product has been criticised
in the state of the art according to EP-A-0 712 703.
Obviously the shortcomings of the crystallisation
device that has been used, with random distribution of
the dwell time, have been recognised in the state of
the art which provides for subsequent heating in a
container comprising rotating circulation devices. As
has been found, it is precisely these circulation
devices which are responsible for generating an
excessive dust fraction. For this reason, with a view
to a more gentle and controlled treatment of the
material, the invention uses a different approach in
that the crystallised material is brought into the
shape of a rectangular bulk material stream of four-
sided, in particular rectangular, cross-section of
essentially even bulking across the cross-section; with
treatment gas flowing from one side of the four-sided

shape through said crystallised material. This means
that as a result of this cross-sectional shape, the
same conditions prevail for the gas along the entire
inflow side, with the essentially even bulk density
contributing its part. This can be improved still
further in that the ratio of the rectangular sides of
the cross-section of the bulk material stream is
approximately 1:2 to 1:15, preferably ranging from 1:3
to 1:10, with the treatment gas being conducted through
the bulk material stream from the larger side of the
rectangle. In this way the method according to the
invention differs from all those methods where the
thicknesses of the bulk material stream and/or the gas
flow conditions along the cross-section are different.

For gentle treatment, precrystallisation and
crystallisation advantageously require a duration of
between 10 and 80 minutes, preferably between 15 and 40
minutes, in particular between approx. 20 and 30
minutes. As mentioned above, according to the
invention, subsequent heating up can be made more
efficient by shortening this treatment step. According
to the present invention this preferably takes place in
that the heating following crystallisation, including
precondensation, is carried out within a duration of 60
to 120 minutes, in particular approx. 90 minutes.

Further details of the invention are provided by means
of a preferred embodiment, diagrammatically shown in
the drawing, as follows:

Fig. 1 is a flow chart of the method according to the
        invention;

Fig. 2 shows a longitudinal section of an embodiment
        of a crystalliser for precrystallisation and

postcrystallisation, according to the
invention.

Fig. 3   shows a cross-section along the line III-III of
         Fig. 2;

Fig. 4   is a diagrammatic drawing with a longitudinal
         section through a device preferably used
         according to the invention, for preheating and
         condensing as well as cooling; and

Fig. 5   shows a cross-section of the above-mentioned
         device according to line V-V of Fig. 4.

According to Fig. 1 in a preliminary stage 1 of the
method according to the invention, amorphous plastic
granulate, in particular polyethylene terephthalate, is
made, i.e. essentially extruded and cut into pellets.
In the first stage of the process according to the
invention, the amorphous plastic material produced in
this way is fed to a crystalliser 2 which
advantageously comprises precrystallisation and
postcrystallisation, as is known from the state of the
art but as will be explained below in Figs. 2 and 3 by
means of an embodiment according to the invention.

The plastic material is at approximately ambient
temperature when it enters stage 2. Within stage 2, gas
treatment with a hot inert gas takes place, such as
nitrogen which is fed into a gas inlet 2' at a
relatively low temperature (when compared to the state
of the art) of approx. 165 °C to 185 °C, e.g. 170 °C to
180 °C so as to achieve gentle treatment of the plastic
material. Accordingly, it is better according to the
invention if the dwell time in the crystalliser 2 is
somewhat longer than is proposed in the state of the
art, namely 10 to 80 minutes, preferably 20 to 40

minutes, in particular approx. 30 minutes. With the relatively low temperature and the relatively long dwell time an even and gentle treatment is achieved and it has been shown that with this method, practically complete crystallisation of the material can be achieved. Tests have shown that of the plastic material leaving stage 2, at the most 1 % has remained amorphous, but generally less than that. Subsequently the final temperature of the plastic material is approx. 135 °C to 180 °C when it leaves stage 2.

The material heated to max. 180 °C must now continue to be heated so as to trigger a condensation reaction. Advantageously, a preheater 3 can be provided for this, followed by the actual reactor 4. As far as the gas circuit 5 (e.g. nitrogen) is concerned, the two stages are interconnected, with a gas purification stage 6 being inserted, before the gas which has passed through the circuit 5 once is again fed to the preheater 3.

The two-stages of the condensation method shown here are normal and advantageous, but not absolutely required. The diagram shown in Fig. 1 merely reflects the fact that it is advantageous if two devices 3 and 4, which are arranged in line, are provided. However, in practical application, preheating where the plastic material is heated to approx. 180 °C (if the material enters at a lower temperature than this) may be carried out separately in a heating device, with the condensation being carried out subsequently in one or several steps. For reasons of efficiency it is however more favourable if preheating and precondensation, and if required also condensation, are combined in one device. For example it would be imaginable to combine preheater or precondenser and condenser (reactor) in a single device. In this case it is advantageous to provide several gas inlets and outlets at various

heights in the vertical reactor so as to divide said
reactor into individual zones of different gas
temperatures and/or gas quantities and/or speeds.

While the reactor 4 will be designed in the
conventional way as a wall bed reactor or packed bed
reactor with a tube, through which the plastic material
passes at a controlled speed, a preferred embodiment of
the preheater 3 will be described later by means of
Figs. 4 and 5. The "controlled speed" within reactor 4
mentioned, can be achieved by installing roof-like
devices extending across the longitudinal axis. Such
roof-like devices exert a braking effect on the
material, thus preventing too rapid a flowthrough, but
by their roof shape which converges to a point at the
top, they promote separation of the individual
particles which per se can have a definite tendency to
stick together.

While the preheater 3 can be designed so as to be in
one stage, it preferably comprises at least two stages,
if necessary up to eight stages, with the gas
temperature advantageously increasing from stage to
stage. Depending on the design, at the end of the
preheater 3, a material temperature of 190 to 235 °C
will result. In the embodiment shown, which comprises
the two stages 3 and 4, a material temperature of
approx. 220 °C will be usual.

Subsequently, the reaction should be terminated as
quickly as possible. For this purpose a cooling
apparatus 7 is connected at the outlet side, with the
postcondensed PET material subsequently issuing from
said cooling apparatus 7. However, the material could
also be a polyolefin, PEN or PA. If precooling takes
place already at the end of the reactor 4, so that the
plastic material issues at a temperature clearly below

185 °C, for example at approx. 160 °C, then it will no longer be necessary for the cooling apparatus 7 to be using inert gas; cooling with air will be possible.

Fig. 2 shows a crystalliser 10 according to the invention which comprises a rotation-symmetrical, in particular cylindrical treatment space 12 (see Fig. 3), surrounded by walls 11, for accommodating the plastic material in the shape of pieces or pellets which issues from the preliminary stage 1 (Fig. 1). For control of precrystallisation with external drying-on of the pellets and crystallisation, said treatment space 12 is divided into at least two treatment compartments 12' and 12". This division is by a partition wall 13. Fig. 3 illustrates that the division is approximately sector-shaped, with the first treatment compartment 12' taking up at least approx. 50 % of the cross-sectional area, preferably 2/3 to 3/4 of the cross-section. By contrast, compartment 12" takes up the remainder of the available space. However, division per se (when seen in top view or in sectional view corresponding to Fig 3) could also take place by means of radial division walls around the longitudinal axis A of the crystallisation apparatus 10. While the embodiment shown only shows two compartments 12', 12", if required, more than two compartments can be provided by at least one further partition wall.

The rotation-symmetrical design ensures a high volume-to-surface ratio, so that not only are heat losses minimal, but also an even temperature in the treatment room 12 is more easily assured. Plastic material can be fed to the treatment room 12 via at least one filling aperture 14. Within the filling aperture, a conventional rotor R for distributing the plastic material during entry in the treatment space can be provided via an upper floor 16'. Due to the relatively

large volume of the compartment 12', or for space
reasons, the wall 13 below preferably comprises a
funnel-shaped deflection section 13' which also imparts
increased rigidity to the wall. Thus, the plastic
material slides down (to the left in Fig. 2) along
section 13', into the first compartment or
precrystallisation compartment 12".

Hot inert treatment gas such as nitrogen, flows through
the treatment space 12. For this purpose a gas inlet
nozzle 15 has been provided. The gas then flows upward
according to the arrows 17, through a perforated floor
16. However, it is understood that as part of the
invention it is quite possible to provide for at least
two gas inlets 15 such that one of them admits gas only
to compartment 12', and the other only to compartment
12". Such a divided gas supply makes it possible if
necessary to treat the plastic material in the two
compartments 12', 12" with differing gas quantities
and/or gas speeds and/or gas temperatures. For example
it might be advantageous to provide for a larger gas
speed in compartment 12', to achieve faster drying of
the surface of the plastic pellets. By contrast, in
compartment 12" perhaps a lower speed but a higher
temperature of the treatment gas is provided.

By providing for a relatively high flow rate of the
gas, and for the treatment space 12 to become enlarged
in an upper section 11' towards the top, both through
the funnel section 13' and through an increase in
diameter of the outer wall 11, the conditions for a so-
called aggregate fluidisation bed are created in which
the plastic material is very strongly fluidised. In
this way, gas flows around the pellets on all sides and
the surface of the pellets is dried. Amorphous pellets
are somewhat lighter than completely crystallised
pellets, and although the difference is not very great,

it contributes to carrying out a selection of still
amorphous and already partially crystallised material,
if the transition from compartment 12' to compartment
12" is selected at the underside of the partition wall
13 where a passage 18 is shown in the drawing. It is
understood that it can for example be advantageous if
the partition wall 13 is connected to the floor 16 by
means of downward protruding stays and if said
partition wall 13 is supported by said floor 16, so
that a number of such free spaces 18 are formed.

The height of the passage depends on the total volume
of the treatment space 12, the degree to which said
treatment space 12 is filled, and the type of material
to be treated. It is thus quite possible within the
scope of the invention, to provide for an adjustment
device for the height of this free space 18, for
example a slide gate which can be approximately slot
shaped, delimiting the lower end of the partition wall
13. If necessary, the vertical position of the wall 13
is secured by radial spokes (not shown), in particular
in the upper part of the treatment space, if need be
also in the lower part. Generally, however, it has been
shown that there is no need for an adjustment device
for adjusting the height of the free space 18, and that
this free space can be fixed. It has been shown that a
gap height for the free space 18, of 3 to 8 cm, in
particular around 5 cm is generally advantageous.

If it is to be possible to operate the crystalliser 10
continuously, as is preferred, an open discharge
aperture 19 must always be provided, said discharge
aperture 19 which according to Fig. 2 is located at the
top of the crystallisation compartment 12" being
provided in the end section of a discharge tube 20
which at its lower end discharges the material to the
exterior. The location of the free space 18 below the

partition wall 13, and the discharge aperture at the top, results in a somewhat meandering or switchback-like path according to the arrows 21 along which the plastic material must flow to travel from the feed aperture 14 to the discharge aperture 19. This not only increases the dwell time in the compartments but it also prevents any "shortcut" which would result if the material were able to pass directly from the free space 18 to a discharge aperture below. As the weight fraction of amorphous material has already dropped below half in compartment 12' (precrystallisation), now only a relatively short treatment in the second compartment 12" (postcrystallisation) is required in order to obtain almost 100 % crystallisation. Therefore, this compartment 12" can be relatively small. A path according to arrows 21 will however also be advantageous if more than one partition wall 13 has been provided, in which case the material after the upward movement in compartment 12", could carry out a downward movement in a third compartment so that the discharge opening could advantageously be located in the floor region.

Fig. 3 shows a few geometric aspects of the design of the crystalliser 10. In top view the gas inlet 15 and a manhole 22 located opposite, result in a transverse axis T along which the gas outlet 23 is situated, as shown in a dot-dash line (in Fig. 2 situated above the sectional plane III-III). So as to provide access via a manhole 22 to both compartments 12' and 12", the compartment 12" is offset by a specified angle α in relation to the transverse axis T through the manhole 22. Said angle can range between 30° and 60° but is essentially not critical. For example 30° has proven advantageous because in such a way a geometric arrangement can be achieved in which the lower section 13" of the partition wall 13 in Fig. 3 is located

approximately in the middle region of the manhole 22.
Since the desired final condition of the plastic
material is to be achieved in compartment 12", it is
preferably associated with at least one monitoring
device, for example a spectrometer "looking" into
compartment 12", said spectrometer detecting the
crystallisation state of the plastic by means of the
spectrogram determined. But at the very least such a
monitoring device can be realised in the shape of an
inspection glass 24. Of course, such a monitoring
device can already be provided in the preceding
compartment, should this be desired.

After passing through the crystalliser (stage 2 in Fig.
1), as shown in the diagram, the plastic material
reaches a preheater 3, a preferred embodiment of which
is shown in Figs. 4 and 5. The arrow 10' in Fig. 4
indicates that at this stage the material emanating
from the crystalliser 10 enters the feed aperture of a
shaft 31 of the preheater (precondenser) 30. Within an
exterior wall 30' approximately circular in shape, the
shaft 31 is of rectangular cross-section, as can be
seen in particular in Fig. 3. The ratio of length L to
width B is for example approximately 1:3 to 1:10,
preferably 1:5 to 1:8, in particular around 1:6. The
shaft 31 is delimited on both sides by means of
perforated plates, sieves or similar 32. This ensures
even flow conditions and treatment conditions along the
entire length L. In addition, the vertical arrangement
of the shaft 31 leads to even density of the material
across the entire cross-section of the shaft.

As is shown, the device 30 which can generally be
called a heat exchanger is divided into several stages
with different functions. If for example N_2 is used as
an inert gas for treatment, said gas can enter at 33 at
a relatively low temperature in order to bring the

treated goods in a compartment 34 separated by an upper
wall 35, to a lower temperature or to expel volatile
compounds which were released in one of the upper
compartments.

After the nitrogen in compartment 34 has streamed
through the sieve walls 32 of the shaft 31, it leaves
this cooling compartment 34 and is subsequently brought
to a higher temperature by an electrical heating device
E. In this, a temperature for example of at least 185
°C is reached which serves as a holding temperature for
precondensation in a compartment 36. Here again the gas
streams in a zigzag through the shaft 31, i.e. in the
opposite direction to the flow in compartment 34 below,
so as to subsequently again be further heated in a
heating device E and to pass through a precondensation
compartment 37 where the plastic material is for
example treated at a gas temperature between 200 °C and
240 °C, if required up to 260 °C. Again, the direction
of flow in this compartment 37 is opposite to that in
the adjacent compartment 36. Subsequently, in the
uppermost compartment 38 after renewed heating, if
required the highest temperature is reached for
preheating the plastic material which is still at a
lower temperature. However, this is not absolutely
essential, for the temperature management can also be
selected such that in the last, uppermost heating
device E, only the temperature losses in the
compartment 36 situated below are compensated for. In
this way the treatment gas essentially flows upwards,
while the plastic material sinks in the shaft 31, thus
moving in reverse flow. The rate of fall in the shaft
31 can be controlled by controlled discharge at the
lower end, for example by means of a cellular wheel
sluice 39 with variable speed or e.g. by way of
temperature sensors at the end of the shaft 31. It is
understood that if required more than four

compartments, e.g. six or eight or fewer, or even only one compartment can be provided, depending on the tasks assigned to the heat exchanger 30, such as only preheating, precondensation, condensation etc. Nor is it necessary for the gas to be led in a zigzag path, but instead, separate gas inlets may be provided for individual compartments, for example to vary the quantity of flow, the type of gas and/or the gas speed in the individual compartments 34 to 38.